

TEST METHOD FOR OXIDIZING SOLIDS

1.0 SCOPE AND APPLICATION

1.1 This method can be used to evaluate the relative oxidizing hazard posed by a solid waste. The method is based on a "conical pile-type burning test method" adapted from the United Nations regulations and classification procedures for the international transportation of dangerous goods.

1.2 This method provides a qualitative means to measure the potential of a solid waste to increase the burning rate or burning intensity of a combustible substance, specifically fibrous cellulose. The burning rates for 1:1 and 4:1 waste:cellulose mixtures (w/w) are compared to the burning rates for 3:7, 2:3, and 3:2 potassium bromate:cellulose mixtures. Potassium bromate is used as the reference oxidizing standard. The three mixing ratios of potassium bromate and cellulose provide a range of burning times by which the waste is classified. The 3:7 mixture ratio provides the longest burning time, the 3:2 mixture ratio provides the shortest burning time, and the 2:3 mixture ratio provides an intermediate burning time. The burning times for the potassium bromate:cellulose mixtures are correlated to their relative oxidizing strengths, i.e., the shorter the burning time, the greater the relative oxidizing strength.

1.3. This test procedure is suitable for assessing the relative oxidizing hazard of solid substances, including solids, granular materials, and other materials that can be formed into a conical pile as described in Sec. 11.2.4. This procedure is not appropriate for evaluating oxidizing liquids. This method may be used to meet certain regulatory applications, but is not required for determining if a waste passes or fails the characteristic of ignitability per the RCRA definition.

1.4 Prior to employing this method, analysts are advised to consult the disclaimer statement at the front of the manual and the information in Chapter Two, Sec. 2.1, for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

2.0 SUMMARY OF METHOD

2.1 Aliquots of the waste sample are mixed thoroughly with fibrous cellulose in both 1:1 and 4:1 weight-to-weight ratios. A 30-g aliquot of the waste:cellulose mixture is formed into a truncated conical pile on a cool, impervious surface by means of a glass funnel having a base approximately 70 mm in diameter. The sample is ignited by means of an electrically-heated wire inserted into the base of the pile. The burning time (in seconds) of each waste mixture is measured under standard conditions and recorded. The test is repeated five times for each waste:cellulose ratio mixture.

2.2 The burning times of the waste mixtures are then compared with the burning time for a standard reference substance consisting of a 3:7 weight-to-weight ratio of potassium bromate and cellulose. If the burning times for both the 1:1 and 4:1 waste mixtures are greater than the burning time for the 3:7 potassium bromate:cellulose standard, then no further testing is required and the results of the test are negative. If one or both of the burning times for the waste mixtures are equal

to or less than that of the 3:7 potassium bromate:cellulose standard, then the waste mixture burning time is compared with the burning times for the 2:3 and/or the 3:2 potassium bromate:cellulose standards.

2.3 This test classifies a solid waste into one of four categories of oxidizers.

2.3.1 Category I: Wastes of either mixture ratio that exhibit a mean burning time less than or equal to the burning time of the 3:2 potassium bromate:cellulose standard are classified in Category I.

2.3.2 Category II: Wastes of either mixture ratio that exhibit a mean burning time less than or equal to the burning time of the 2:3 potassium bromate:cellulose standard and greater than the burning time of the 3:2 potassium bromate:cellulose standard are classified in Category II.

2.3.3 Category III: Wastes of either mixture ratio that exhibit a mean burning time equal to or less than the burning time of the 3:7 potassium bromate:cellulose standard and greater than the burning time of the 2:3 potassium bromate:cellulose standard are classified in Category III.

2.3.4 Category IV: Wastes of either mixture ratio that do not ignite and burn, or that exhibit a burning time for both mixture ratios greater than the burning time of the 3:7 potassium bromate:cellulose standard, are classified in Category IV.

3.0 DEFINITIONS

The following definitions are intended solely as guidance to assist the user in properly classifying wastes that have oxidizing properties.

3.1 Oxidizing substance - Any substance which, while in itself is not necessarily combustible, may generally yield oxygen and cause, or contribute to, the combustion of other material.

3.2 Burning rate - For the purpose of this test, the burning rate is defined as the time, in seconds, from the application of electrical power to the heating wire until the main reaction (e.g., flame, incandescence, or glowing combustion) ends. Intermittent reactions, such as sparking or sputtering after the main reaction is completed, are not considered.

4.0 INTERFERENCES

This method measures the burning rates of various mixtures of potassium bromate and cellulose which are used as standards to categorize waste mixtures under a set of uniform experimental conditions. Although no interferences have been reported for this method, several factors such as particle size, reagent moisture content, room temperature, humidity, ventilation, position of the test sample in the hood, and the position of the ignition wire within the test pile can have a significant impact on the burning rates and overall method precision. In order to obtain reliable and reproducible results, it is essential that all steps be conducted in a consistent manner under the same experimental conditions. Strict adherence to all reagent specifications (i.e., particle

size, moisture content, etc.) must be observed and the test conditions must be held constant throughout the evaluation. Refer to Sec. 11.0 for more information.

5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 This method could involve the combustion of highly flammable materials and the generation of toxic fumes. All tests must be conducted in a suitable fume hood fitted with a pull-down sash to prevent the escape of toxic fumes into working areas. The analyst should wear appropriate protective clothing, including a laboratory jacket or apron, safety glasses, and protective gloves. The laboratory should have appropriate fire fighting equipment (such as a Class A fire extinguisher) readily available to extinguish small fires.

6.0 EQUIPMENT AND SUPPLIES

6.1 An ignition source comprising of an inert metal wire such as platinum or nickel which can be electrically heated to approximately 1,000°C. The wire is formed into the shape shown in Figure 1.

6.2 Low-heat conducting, non-combustible, impervious ceramic tile or equivalent material of sufficient size to support a 70-mm diameter test sample. This tile will be used as a platform to conduct the burning rate tests for the reference substances and waste mixtures.

6.3 A thermocouple to measure the temperature of the ignition wire.

6.4 A calibrated thermometer to measure room temperature.

6.5 Standard sieves, 0.150-mm, 0.300-mm, and 0.500-mm (Tyler screen scale equivalent of 100-, 48-, and 32-mesh respectively).

6.6 A 60° glass funnel, sealed at the narrow end, with an internal diameter of 70 mm. This funnel is used to form the reference standards and waste mixtures into truncated conical piles with a 70-mm base.

6.7 Stopwatch capable of measuring 0.1 second increments.

6.8 500-mL glass beakers or other containers suitable for preparing the reference standards.

7.0 REAGENTS AND STANDARDS

Reagent grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used,

provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Potassium bromate (KBrO_3), crystalline (CASRN 7758-01-2).

NOTE: Potassium bromate must have a nominal particle size range of 0.15 to 0.30 mm. It should be sieved through the 0.30-mm sieve followed by the 0.15-mm sieve. Light crushing and grinding is permissible to achieve the desired particle size if necessary. Carefully dry these reference substances at $65 \pm 2^\circ\text{C}$ for 12 hours and store in a desiccator until used.

7.2 Cellulose fibers, with a fiber length between 50 and 350 μm and a mean diameter of 25 μm , to be used as a combustible substrate with the reference reagent and samples. Dry in a layer no more than 25 mm in thickness at $105 \pm 5^\circ\text{C}$ for 4 hours and store in a desiccator until required. Each batch should be used within 24 hours of its preparation. The water content should be less than 0.5% by dry mass.

7.3 Reference standards

7.3.1 Potassium bromate and cellulose reference standard (3:7) - Each reference standard is tested five times to determine the mean burning time. Each trial is conducted with 30 ± 0.1 g of mixture. In a 500-mL beaker or other suitable glass container, mix 60 g of potassium bromate (Sec. 7.1) and 140 g cellulose (Sec. 7.2). The materials must be thoroughly mixed to assure a homogeneous mixture. Mechanical mixing devices may be used to assure complete mixing. The reference substance should be stored in a desiccator until the test is conducted. The 200 g of reference material will provide enough material for five trials using 30 ± 0.1 g of material, plus an additional 30-g aliquot of material, if needed. This reference standard has the longest burning time.

7.3.2 Potassium bromate and cellulose reference standard (2:3) - Prepare in the same manner as described in Sec. 7.3.1, except that 80 g of potassium bromate are mixed with 120 g of cellulose. This reference standard has an intermediate burning time.

7.3.3 Potassium bromate and cellulose reference standard (3:2) - Prepare in the same manner as described in Sec. 7.3.1, except that 120 g of potassium bromate are mixed with 80 g of cellulose. This reference standard has the shortest burning time.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Sample collection

8.1.1 Samples should be collected in clean glass containers fitted with PTFE-lined screw caps.

8.1.2 This procedure requires a minimum sample weight of approximately 260 g. It is recommended that more than 500 g of sample be collected to allow for retesting or confirmation of initial test results, if necessary.

8.2 Sample preservation - No preservatives are added to the sample. The waste is tested at ambient laboratory temperature.

8.3 Sample storage - Samples should be stored at $4 \pm 2^{\circ}\text{C}$ until evaluated. Prior to conducting this test, the sample should be allowed to equilibrate to ambient laboratory temperature ($20 \pm 5^{\circ}\text{C}$). If the loss of volatile organic constituents are of concern, the entire sample in its original sample container should be allowed to equilibrate to ambient laboratory temperature.

8.4 A holding time for oxidizing solids has not been established. The sample should be tested as soon as possible to avoid any further chemical or physical changes.

9.0 QUALITY CONTROL

9.1 The mean burning time for each reference standard is determined using a minimum of five separate trials each time the test is performed. Perform each evaluation as described in Sec. 11.0 and average the results of the trials. It is not necessary to determine the burning time for all three reference substances as long as the waste can be properly categorized (Sec. 2.3). For example, if a waste does not ignite or has a burning time greater than that of the 3:7 potassium bromate:cellulose standard, no further testing is required and the burning times for the 2:3 and 3:2 potassium bromate:cellulose standards need not be determined. Should the waste exhibit a burning time less than the 3:7 potassium bromate:cellulose standard, prepare and test the 2:3 and/or 3:2 potassium bromate:cellulose reference standards as needed.

9.2 Each waste is evaluated in 1:1 and 4:1 weight-to-weight (waste:cellulose) ratios. The burning time for each waste mixture ratio is evaluated in five separate trials and the average burning time is calculated for each waste mixture ratio by dividing the sum of the burning times by five.

9.3 Each waste is categorized based on the shortest mean burning time for either waste mixture. For example, if the mean burning time for the 1:1 waste mixture is 55 seconds and the mean burning time for the 4:1 waste mixture is 28 seconds, then the 28 second burning time is compared to the mean burning times exhibited by the reference standard(s) in order to make the appropriate oxidizing classification.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Determine the mean burning time (in seconds) for each reference standard using a minimum of five separate trials according to the procedures contained in Sec. 11.0. The mean burning times for the standard(s) must be determined each time the test is performed. The mean burning time for each reference standard is obtained by dividing the sum of the five burning trials by five.

10.2 The burning time for all three standard reference mixtures need not be determined, as long as the waste can be properly categorized, as described in Sec. 2.3.

11.0 PROCEDURE

11.1 Waste preparation

11.1.1 The waste to be evaluated is tested in its "as received" form and should not be dried. As noted in Sec. 8.3, the waste should be stored at $4 \pm 2^{\circ}\text{C}$. It should be allowed to equilibrate to ambient laboratory temperature ($20 \pm 5^{\circ}\text{C}$) immediately prior to analysis.

11.1.2 Evaluate the particle size of the waste, in order to assure consistent burning rate times and to assure the complete mixing of waste and cellulose. The waste should be cut, crushed, or ground so that the particle size of the sample to be tested is no larger than 0.5 mm (passes through a 32-mesh sieve).

11.1.3 Waste:cellulose mixture (1:1) - In a 500-mL beaker or other suitable glass container, mix 100 g of waste and 100 g of cellulose (Sec. 7.2). The materials must be thoroughly mixed to assure a homogeneous mixture. Mechanical mixing devices may be used to assure complete mixing. This will provide enough test material to conduct five burning rate trials using 30 ± 0.1 g of material per trial, and an additional aliquot if needed.

11.1.4 Waste:cellulose mixture (4:1) - In a 500-mL beaker or other suitable glass container, mix 160 g of waste and 40 g of cellulose using the procedure described in Sec. 11.1.3.

11.2 Burning rate test

11.2.1 Weigh five separate 30.0 ± 0.1 g aliquots of the 3:7 potassium bromate:cellulose reference standard (Sec. 7.3.1) into five separate glass beakers or other suitable containers.

11.2.2 Position the ceramic tile (Sec. 6.2) in a fume hood approximately 20 cm (8 inches) from the front of the hood in an area of laminar airflow. Air flow across the test sample should be minimal (approximately 0.5 - 0.7 m/s), but sufficient to prevent the escape of fumes or smoke into working areas.

11.2.3 Position the ignition wire over the center of the ceramic tile. The ignition wire is formed into a 40-mm circular loop (see Figure 1), approximately 1 mm above the test surface.

11.2.4 Place $30.0 \text{ g} \pm 0.1 \text{ g}$ of reference substance into the glass funnel. Tap the funnel gently to settle the material in the funnel, place the ignition wire and ceramic tile on top of the funnel, and then invert the funnel and sample to cover the ignition wire and ceramic tile. Remove the funnel. The reference substance should be in the form a truncated conical pile with a base approximately 70 mm in diameter, centered over the ignition wire so that the wire is completely covered by the test sample. Refer to Figure 1 for placement of the conical pile and ignition wire.

11.2.5 Measure the burning time of the 3:7 potassium bromate:cellulose reference standard by turning on the electric power and heating the ignition wire to at least 1000°C , but no greater than 1050°C . Begin timing when the power is applied to the wire and continue timing until the end of the main combustion. Once the pile is ignited, turn off the power to the ignition wire. Record the total burning time in seconds. Repeat the burning time trial four

more times, using a fresh $30.0 \text{ g} \pm 0.1$ gram aliquot of reference standard for each trial, and calculate the mean burning time. Allow the ceramic tile to cool to ambient temperature between time trials. In order to ensure consistent data, all tests must be conducted under standard test conditions, which include a temperature of 20 ± 5 °C and a relative humidity of $50 \pm 10\%$.

11.2.6 Measure the burning time for each of five aliquots of the 2:3 potassium bromate:cellulose standard (Sec. 7.3.2) and the 3:2 potassium bromate:cellulose (Sec. 7.3.3) standard as described in Secs. 11.2.1 through 11.2.5. Calculate the mean burning time for the standards. Note that, depending on the burning times exhibited by the waste mixtures, a determination of the burning times for all three reference mixtures may not be required. Refer to Sec. 9.1.

11.2.7 Measure the burning times of the 1:1 and 4:1 waste:cellulose mixtures as described in Secs. 11.2.1 through 11.2.5. Each waste concentration is evaluated with five separate time trials and the average burning time is calculated. The shortest mean burning time for either waste concentration is used to categorize the waste.

11.2.8 Apply power to the ignition wire as described in Sec. 11.2.5 until observable signs of combustion are evident. If the waste mixture does not appear to ignite, continue heating the ignition wire for a period of at least three minutes. If the waste fails to ignite after this period of time, no additional trials are required and the results of the oxidizing test are negative.

NOTE: This procedure compares the mean burning time exhibited by a waste to that of standard reference materials having known burning characteristics. It is therefore critical that the test be conducted in a consistent manner in order to assure reliable and consistent results. Care should be taken to assure that all test conditions are held constant to the extent possible. The position of the ceramic tile within the fume hood and the position of the ignition wire within the conical pile should be the same for all trials. Recognition of ignition and termination of burning must be applied in a consistent manner for both reference substances and waste mixtures.

11.3. Criteria and method for assessing results

The results are assessed on the basis of:

- whether the waste mixture ignites and burns; and
- the comparison of the average burning time for the fastest of the two (2) waste mixtures with those of the reference standards.

12.0 DATA ANALYSIS AND CALCULATIONS

A waste sample is categorized by comparing the shortest mean burning time for either of the waste mixtures (1:1 waste:cellulose and 4:1 waste:cellulose) with the mean burning times exhibited by the standard reference substances. Category I wastes have the highest oxidizing strength, while wastes in category III have the lowest oxidizing strength. Wastes that do not ignite or burn within three minutes, or have burning times greater than the 3:7 potassium bromate standard, are not

considered oxidizers under this test, and are placed in category IV. Use the table below to make the appropriate classification.

If the Shortest Mean Burning Time in Seconds for Any Waste Mixture Is...	Then the Waste Is:
Less than or equal to that of the 3:2 potassium bromate:cellulose standard	Category I
Less than or equal to that of the 2:3 potassium bromate:cellulose standard and greater than that of the 3:2 potassium bromate:cellulose standard	Category II
Less than or equal to that of the 3:7 potassium bromate:cellulose standard and greater than that of the 2:3 potassium bromate:cellulose standard	Category III
Greater than that of the 3:7 potassium bromate:cellulose standard or does not ignite or burn within three minutes	Category IV (a non-oxidizer)

13.0 METHOD PERFORMANCE

13.1 In an evaluation of the standard reference oxidizer/fuel mixtures, the burning rates for the 3:7, 2:3, and 3:2 (w/w) potassium bromate:cellulose mixtures were determined. The mean burning times and standard deviations exhibited by each of the three reference mixtures are summarized in Table 1. Separate aliquots of 30 ± 0.1 g of each mixture were formed into conical piles and ignited by means of an electrically-heated ignition wire. Each reference mixture was tested with a minimum of six separate trials. These data are provided for illustrative purposes only, and the actual burning times obtained by the laboratory may vary, depending on the specific experimental conditions (see Reference 4).

13.2 Example burning times of two sample mixtures and the three standards are given in Table 2. All data are taken from Reference 5. A comparison of the data in Tables 1 and 2 reveals that the absolute burning time for the standards can differ significantly between laboratories. The cellulose, the potassium bromate, the preparation steps, and the ambient laboratory conditions are all contributing factors to the burning time. However, a sample is not classified according to its absolute burning time, but rather, based on its burning time relative to the standards analyzed each time that samples are tested. Thus, even though there will be differences in burning times between laboratories, the procedure will yield correct classifications of waste samples.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of a waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques

to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction* available from the American Chemical Society, 1155 16th Street NW, Washington D.C., 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Hasegawa et al, "Characteristics of the Conical Pile Type Burning Test Method for Determining the Potential Hazards of Oxidizing Material", *J. Loss. Prev. Process Ind.*, 1989, Vol 2, July, p 135.
2. Title 49, Part 173, Appendix F, Guidelines for the Classification and Packaging Group Assignments of Division 5.1 Materials.
3. United Nations, Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria, ST/SG/AC.10/11/Rev 2, Section 34, Classification Procedures, Test Methods and Criteria Relating To Oxidizing Substances of Division 5.1, Test O.1: *Test for Oxidizing Solids*, 1995.
4. Science Applications International Corporation, Final Report for the Evaluation of Method 1040, submitted to the US EPA, Office of Solid Waste, June 1998.
5. Fowler, John; Personal Communication from John Fowler, NEIC, to Oliver Fordham, OSW, July 1999.

17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 and 2, Figures 1, and a flow diagram of the method procedure.

TABLE 1

EXAMPLE MEAN BURNING TIMES (IN SECONDS) AND STANDARD DEVIATIONS
OF OXIDIZER/FUEL MIXTURES

Oxidizer:Fuel Ratio	Mean Burning Time in Seconds \pm SD
3:7 Potassium Bromate:Cellulose	64 \pm 14.8 ¹
2:3 Potassium Bromate:Cellulose	28 \pm 8.2 ²
3:2 Potassium Bromate:Cellulose	16 \pm 0.7 ²

¹ Results based on six trial burns.

² Results based on seven trial burns.

All data are taken from Reference 4 and are provided for illustrative purposes only. The burning times for the standard mixtures must be determined each time the test is conducted.

TABLE 2
EXAMPLE BURNING TIMES OF VARIOUS MIXTURES

Test No.	Burning Time (seconds)				
	Sample/Cell. 4:1	Sample/Cell. 1:1	KBrO ₃ /Cell. 3:7	KBrO ₃ /Cell. 2:3	KBrO ₃ /Cell. 3:2
1	11	38	105	58	10
2	14	--	105	50	11
3	10	--	129	56	8
4	13	--	103	56	12
5	14	--	118	60	10
Mean	12	38	112	56	10
Std. Dev.	1.8	--	11.2	3.7	1.5
%RSD	14.6	--	10.0	6.7	14.8

Conclusion: Since the 4:1 sample/cellulose mixture burned at a rate between the 2:3 and 3:2 KBrO₃/cellulose standards, the sample material is a DOT 5.1 oxidizer and falls within Packing Group II.

Subsequent chemical analysis of the sample found that it was approximately 55% sodium chlorate with the remaining material being mostly sodium chloride. Sodium chlorate is a known oxidizer and in its pure form is listed in Packing Group II.

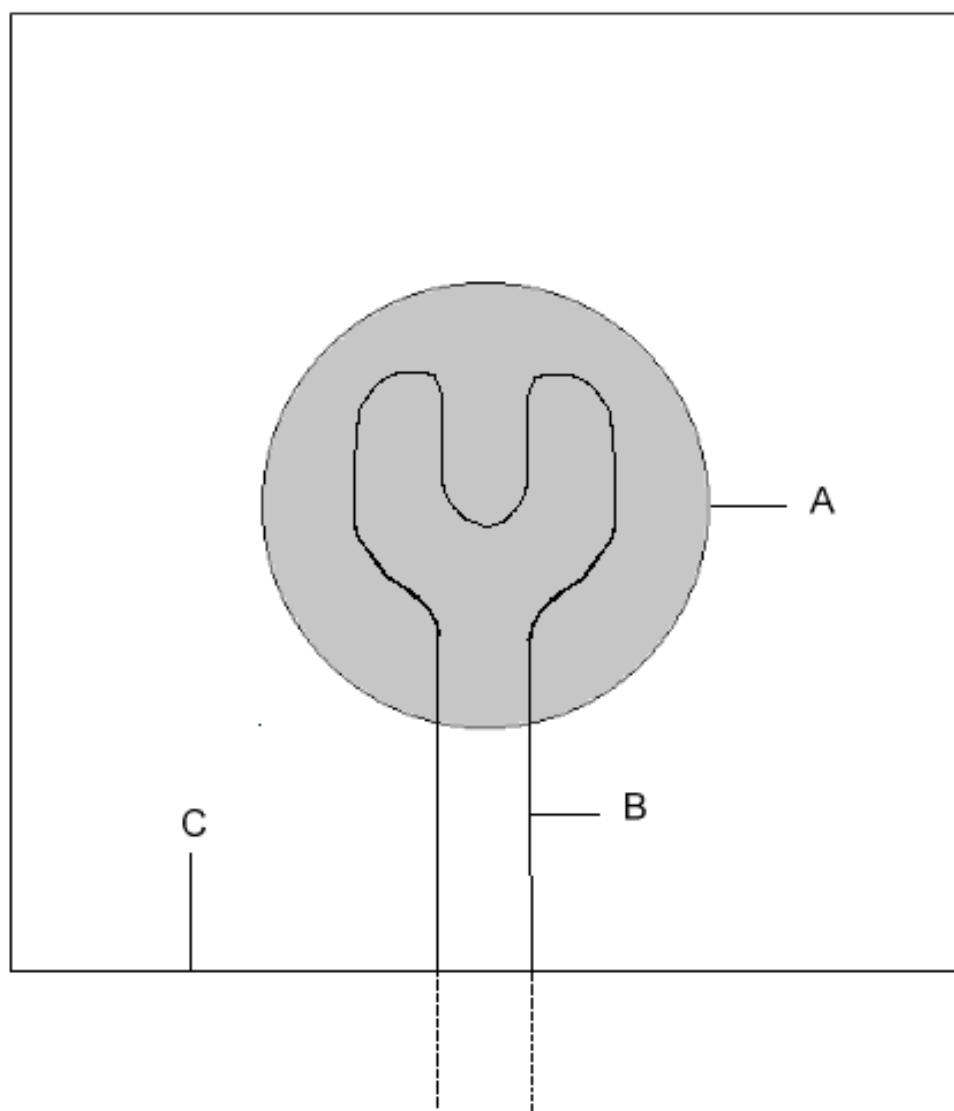
All data are taken from Reference 5 and are provided for illustrative purposes only. The burning times for the standard mixtures must be determined each time the test is conducted.

For comparison, the DOT reference values for the standards are:

3:7 KBrO ₃ /cellulose	100 seconds
2:3 KBrO ₃ /cellulose	54 seconds
3:2 KBrO ₃ /cellulose	4 seconds
4:1 pure sodium chlorate/cellulose	5 seconds

FIGURE 1

Test Plate and Ignition Wire
(not to scale)



- A = Outline of the base of the 70-mm conical pile
- B = Approximate position and shape of the ignitor wire
- C = Ceramic tile or other non-combustible material, approximately 150 mm on each side

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